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AN ACETYLATION METHOD FOR DETERMINING MIXTURES OF EITHER HYDRAZINE-1,1-DIMETHYLHYDRAZINE OR MONOMETHYLHYDRAZINE-1,1-DIMETHYLHYDRAZINE

TECHNICAL DOCUMENTARY REPORT NO. SSD-TDR-62-203

DECEMBER 1962

ROCKET RESEARCH LABORATORIES
CHEMICAL AND MATERIALS BRANCH
EDWARDS, CALIFORNIA

HEADQUARTERS
SPACE SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE

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ABSTRACT

A method is presented for determining mixtures of either hydrazine-1, 1-dimethylhydrazine (N_2H_4/UDMH) or monomethylhydrazine-1, 1-dimethylhydrazine (MMH/UDMH). This method is based on the difference in reaction rates for acetylating hydrazines. Both N_2H_4 and MMH react immediately while the acetylation of UDMH proceeds slowly. The reactions are conducted in acetonitrile-acetic acid medium. Two titrations are required: one measures the total basicity of the hydrazine mixtures; the other titration measures UDMH after the N_2H_4 or MMH has reacted with Ac_2O.

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INTRODUCTION.

Hydrazine (N_2H_4), monomethylhydrazine (MMH), and 1, 1-dimethylhydrazine (UDMH) are important both individually and in mixtures as rocket fuels. A 50-50 percent mixture of N_2H_4/UDMH is used as the fuel blend for the Titan II. Though mixtures of MMH/UDMH are discussed herein, they are not presently being used in any weapon system.

The purpose of this report is to present a method for determining mixtures of either N_2H_4/UDMH and MMH/UDMH based on observed differences in reaction times with acetic anhydride.

BACKGROUND.

Several methods have been developed for analysis of mixtures of hydrazine-1, 1-dimethylhydrazine. These methods use gas chromatographic techniques (1, 5), standard iodate titration in an aqueous medium (9), or the selective reaction of hydrazine with salicylaldehyde and subsequent titration of basicity with perchloric acid in non-aqueous medium (6). This latter method is presently used in Specification MIL-P-27402. It has the end point disadvantage encountered in most weak base-strong acid titrations.

Acetic anhydride used herein for acetylating hydrazines was used primarily as a solvent for titrations of amides (10), weak bases (3), and nitrogen bases (8). It has been used also to improve the end point in non-aqueous titrations (10). Fritz and Schenk (4) used acetic anhydride for acid catalyzed acetylation of hydroxyl compounds in ethyl acetate.

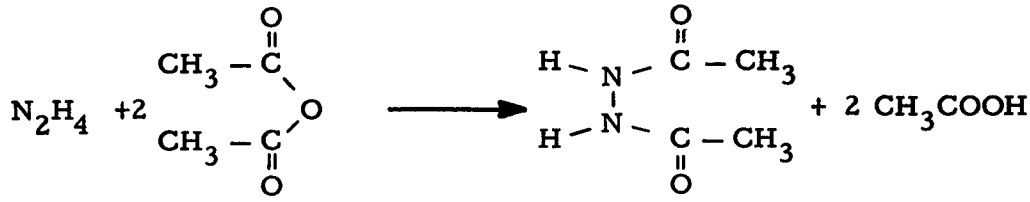
Although the reactions for acetylating hydrazines are known (2) and reported (7), the authors are unaware of any efforts to determine hydrazine and substituted hydrazines by this method.

EXPERIMENTAL.

Reactions of hydrazine, monomethylhydrazine and 1, 1-dimethylhydrazine with acetic anhydride. Reaction products are shown in the following equations:

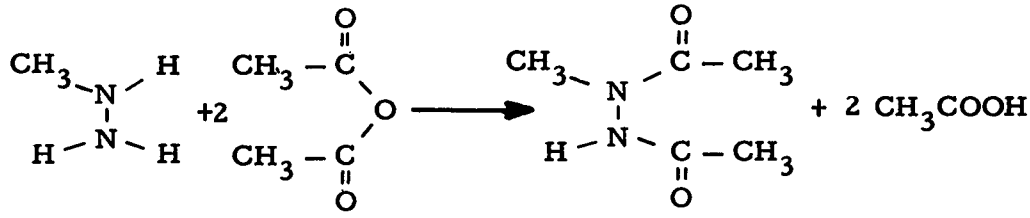
With N_2H_4-1, 2 diacetylhydrazine:

Equation (1)



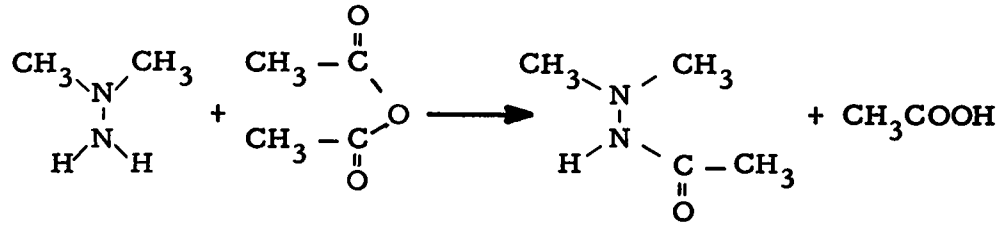
With MMH-Monomethyl 1, 2 diacetylhydrazine:

Equation (2)



With UDMH-1, 1-Dimethyl 2 acetylhydrazine:

Equation (3)



The rates of the reaction differ greatly as both the N_2H_4 and MMH are rendered neutral to perchloric acid titration almost immediately upon UDMH

reacts slowly in acetic acid medium. The product of equation (3) occurs only after a considerable time lapse at room temperature. Figure I shows the effect of time and temperature on the UDMH-acetic anhydride reaction. The reaction, as indicated by a decrease in milliters of $HClO_4$ is slow until the temperature is increased.

Effect of Acetic Anhydride.

As the quantity of acetic anhydride is increased in samples of N_2H_4, MMH, and UDMH, the milliliters of $HClO_4$ titrant required for neutralization decreases much faster with N_2H_4 and MMH. The UDMH is slow to react even when a considerable excess of acetic anhydride is added. This effect is shown in Figure II. These curves were plotted from titration, with perchloric acid, of individual samples containing a 1 ml aliquot of each hydrazine compound per 50 ml acetic acid. Duplicate samples were treated with 1 ml aliquots of acetic anhydride samples containing 1.0 ml - 6.0 ml acetic anhydride in 50 ml acetic acid. The two curves for both MMH and N_2H_4 substantiate the molar ratio presented in equation (1) and (2).

An analysis, after acetylation, of the individual hydrazines taken from stored drums is shown in Table I.

TABLE I.

CORRECTION VALUES FOR $HClO_4$ TITRATIONS OF HYDRAZINES

Sample No.	N_2H_4 ml $HClO_4$ after Ac_2O	UDMH ml $HClO_4$ after Ac_2O[1]	MMH ml $HClO_4$ after Ac_2O
1	0.20	0.04	0.14
2	0.23	0.03	0.10
3	0.20	0.06	
4	0.21		
5	0.20		
6	0.17		
7	0.05		

[1]Ml $HClO_4$ less than total alkalinity value. Sample size 0.025 g.

N_2H_4 drums 1, 2, and 3 were stored for over five years, while drums 4 and 6 were stored for over two years. Drum 5 was stored only six months. Sample marked 7 was redistilled over a 75 plate distillation column. The UDMH and MMH drums were stored for one year.

The values presented above result from the presence of contaminants in the hydrazines. Aniline and ammonia are the usual titratable contaminants in N_2H_4. Nitroso-dimethylamine, ammonia, dimethylamine, methylene dimethylhydrazine, and methylhydrazine are the titratable contaminants in UDMH (12). Of these contaminants, aniline and methylhydrazine react similarly to N_2H_4 and are nontitratable after acetylation. Methylene dimethylhydrazine, dimethylamine, ammonia, and nitrosodimethylamine will be acetylated at different rates and could titrate as UDMH. Further work is necessary to establish the rates of reaction for these contaminants. However, their presence in the hydrazines are not significant except for dimethylamine (0.7%) and nitrosodimethylamine (0.3%) in UDMH and ammonia (0.3%) in N_2H_4.

The contaminants usually present in MMH have not been identified as yet. Until they are, titrations of the individual hydrazines are necessary.

Preliminary observations using other acetylation compounds such as acetyl chloride, benzoyl chloride, maleic anhydride, and benzoic anhydride with UDMH indicate that only the two anhydrides can be used as possible substitutes for acetic anhydride.

Effect of Water.

The water content in N_2H_4-UDMH samples was increased from 0.99% to 7.84% and the samples were analyzed by the procedure presented. Table II shows that increasing the water content does not interfere with the titration.

TABLE II

Percent Water	Sample Weight N_2H_4/UDMH before H_2O added	Total Weight of Sample	ml $HClO_4$ Total Alkalinity	ml $HClO_4$ after Acetylation
0.99	0.8918	0.9007	4.24	1.42
2.31	0.8913	0.9124	4.24	1.42
4.65	0.8903	0.9337	4.24	1.42
7.84	0.8818	0.9570	4.19	1.40

The large excess of acetic anhydride in the N_2H_4 reaction reacts with any water to form acetic acid and eliminates the water interference problem encountered in most non-aqueous titration procedures.

Potentiometric and Colorimetric Endpoints.

Both the potentiometric and quinaldine red indicator endpoints were evaluated. A Beckman zeromatic pH meter with glass-calomel electrodes was used to determine the stoichiometric endpoints for both the total alkalinity titration and the acetic anhydride titration. Figure III shows a typical curve obtained in these titrations. The quinaldine red endpoint changes from red (actually pink) to colorless approximately 0.01 ml after the greatest change in potential occurs, dictated by the millivolt change plot shown in Figure IV. The indicator and potentiometer endpoints can be improved appreciably by using acetonitrile as the solvent in both titrations. For the plots shown an acetonitrileacetic acid mixture (4:1 V/V) was used.

The endpoints can also be greatly improved by using either dioxane or nitromethane as solvents. However, we have encountered considerable difficulty in obtaining dioxane with a low blank value. The nitromethane is such a good monopropellant that we avoid using it as a solvent except when absolutely necessary.

PROCEDURE.

Preparation of Sample.

Pipette 0.4 ml of the N_2H_4-UDMH mixture into a tared, cooled 50-ml volumetric flask containing 40 ml of acetic acid. Weigh to the nearest 0.1 mg, obtaining the sample weight by difference. Dilute to the 50-ml mark with acetic acid and mix thoroughly.

Determination of the Total Basicity of the Hydrazine-UDMH Mixture - Pipette a 5 ml aliquot from the prepared sample into a 50-ml beaker containing 20 ml of acetonitrile. Add 5 drops of quinaldine red indicator and titrate with 0.1 N perchloric acid in acetic acid until the red color of the indicator changes to colorless. Indicate this amount for value "A" in calculations. Titrate a blank of acetonitrile and quinaldine red indicator to the colorless endpoint.

Determination of 1, 1-Dimethylhydrazine - Pipette a 5 ml aliquot of the prepared sample into a 50-ml beaker containing 20 ml of acetonitrile-acetic acid (4:1 V/V). Add 2 ml of acetic anhydride and 5 drops of quinaldine red indicator. Titrate with 0.1 N perchloric acid in acetic until the red color of the indicator changes to colorless. Indicate this amount for value "B" in calculations. Titrate a blank of acetonitrile-acetic acid, acetic anhydride and quinaldine red indicator to the colorless endpoint.

CALCULATIONS.

$$\% \text{ Hydrazine} = \frac{(A-a) - (B-b) \times N \times M_1}{w} \times 10$$

$$\% \text{ UDMH} = \frac{(B-b) \times N \times M_2}{w} \times 10$$

$$\% H_2O = 100 - (\% \text{ Hydrazine} + \% \text{ UDMH})$$

where:

A = ml perchloric acid for total basicity.

B = ml perchloric acid for the anhydride addition.

a = ml perchloric acid for blank of acetonitrile and indicator.

b = ml perchloric acid for blank of acetonitrile-acetic acid, acetic anhydride and indicator.

N = normality of perchloric acid.

M_1 = milliequivalent weight of hydrazine (0.03205).

M_2 = milliequivalent weight of UDMH (0.06010).

w = weight of sample

REAGENTS

Acetic acid - glacial reagent grade.

Acetic anhydride - ACS reagent grade.

Acetonitrile - ACS reagent grade.

Quinaldine red - 0.2 percent in acetic acid.

0.1 N perchloric acid in acetic acid - dissolve 8.5 ml of 72% perchloric acid in 1 liter of glacial acetic acid and standardize against primary reagent potassium acid phthalate.

RESULTS.

Several samples of N_2H_4-UDMH mixtures were prepared and analyzed. Table III presents the theoretical, experimental, and variation from calculated percent obtained for samples ranging from 10% N_2H_4-90% UDMH to 90% N_2H_4-10% UDMH.

TABLE III

Experimental		Theoretical		Variation in %	
N_2H_4	UDMH	N_2H_4	UDMH	N_2H_4	UDMH
92.18	7.58	92.31	7.33	-0.13	+0.23
96.34	13.03	86.61	13.05	-0.27	-0.02
73.98	25.43	74.31	25.38	-0.33	+0.05
65.59	34.36	65.29	34.67	+0.30	-0.31
55.99	44.35	55.97	44.03	+0.02	+0.32
57.79	42.03	58.18	41.82	-0.39	+0.21
55.12	45.13	55.20	44.80	-0.08	+0.33
55.77	43.80	55.91	44.09	-0.14	-0.29
50.40	48.05	50.30	48.25	+0.10	-0.20
20.65	78.36	20.25	78.80	+0.40	-0.44
12.20	87.63	11.69	88.17	+0.51	-0.54

Standard Deviation N_2H_4 0.28
UDMH 0.32

The percent error is greater for samples containing larger UDMH content. This can be attributed to the fact that most of the impurities in the mixtures will react similarly to UDMH. In the above samples of N_2H_4/UDMH mixtures, the correction values were not used. The correction values for both the N_2H_4 and UDMH were 0.04 ml.

Several samples of MMH-UDMH mixtures were prepared and analyzed.

Table IV presents the theoretical, experimental, and variation from calculated value.

TABLE IV

Experimental		Theoretical		Variation in %	
MMH	UDMH	MMH	UDMH	MMH	UDMH
41.56	56.54	41.73	57.04	-0.17	-0.50
50.42	49.24	51.06	48.19	-0.64	+1.05
40.07	55.88	40.47	56.06	-0.40	-0.18
47.01	49.84	47.64	51.19	-0.63	-0.35
62.97	34.47	63.12	34.92	-0.15	-0.45
63.57	36.07	63.89	35.34	-0.32	+0.73

Standard Deviation MMH 0.43
UDMH 0.61

When the correction values discussed in Table I are made to MMH/UDMH samples, the following results are obtained:

TABLE V

Experimental		Theoretical		Variations in %	
MMH	UDMH	MMH	UDMH	MMH	UDMH
41.85	57.10	41.73	57.04	+0.12	+0.06
50.78	48.06	51.06	48.19	-0.18	0.13

We encounter difficulty in the analysis of the hydrazine mixtures when the correction values for the individual hydrazines are unknown. The correction values are applied by equating the value for the individual hydrazine proportional to the total concentration hydrazines in the mixture. The UDMH correction value (resulting from MMH type contaminants) is added to the milliliters acid required for either the N_2H_4 or MMH titration. The N_2H_4 or MMH correction value (resulting from UDMH type contaminants) is subtracted from the milliliters acid required for the UDMH titration.

When the correction values become excessive, the results will be unsatisfactory. Values this high are not usually encountered in stored hydrazines. We presume that the high-value MMH samples reported previously were contaminated accidentally and are not representative correction values normally encountered.

SUMMARY.

Detailed procedures for potentiometric or colorimetric titration in acetonitrile/acetic acid solvent with perchloric acid titrant are presented. Mixtures of either UDMH/N_2H_4 or UDMH/MMH can be analyzed in this system. The procedural basis is the difference in reaction rates for acetylating hydrazines with acetic anhydride.

Analysis of N_2H_4/UDMH mixtures gave standard deviations of 0.28 for N_2H_4 and 0.32 for UDMH. Standard deviations found for analysis of MMH/UDMH were 0.43 for MMH and 0.61 for UDMH. The standard deviation decreases when correction values are applied.

RECOMMENDATION.

The acetylation procedure for analysis of N_2H_4/UDMH is recommended to supersede the aldehyde procedure currently required in Specification MIL-P-27402.

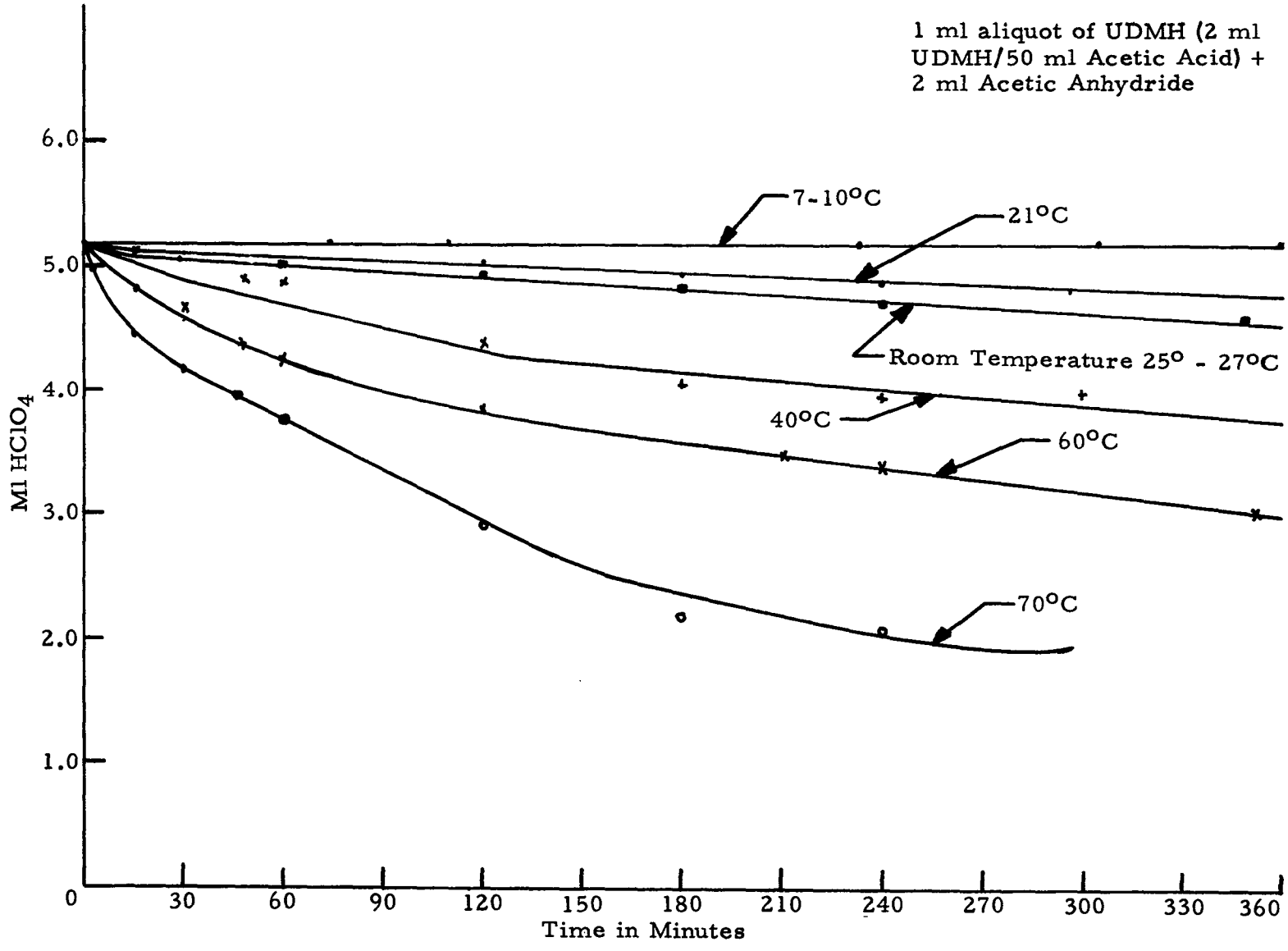


Figure I. Effect of Time and Temperature on the Reaction Rate of UDMH with Acetic Anhydride.

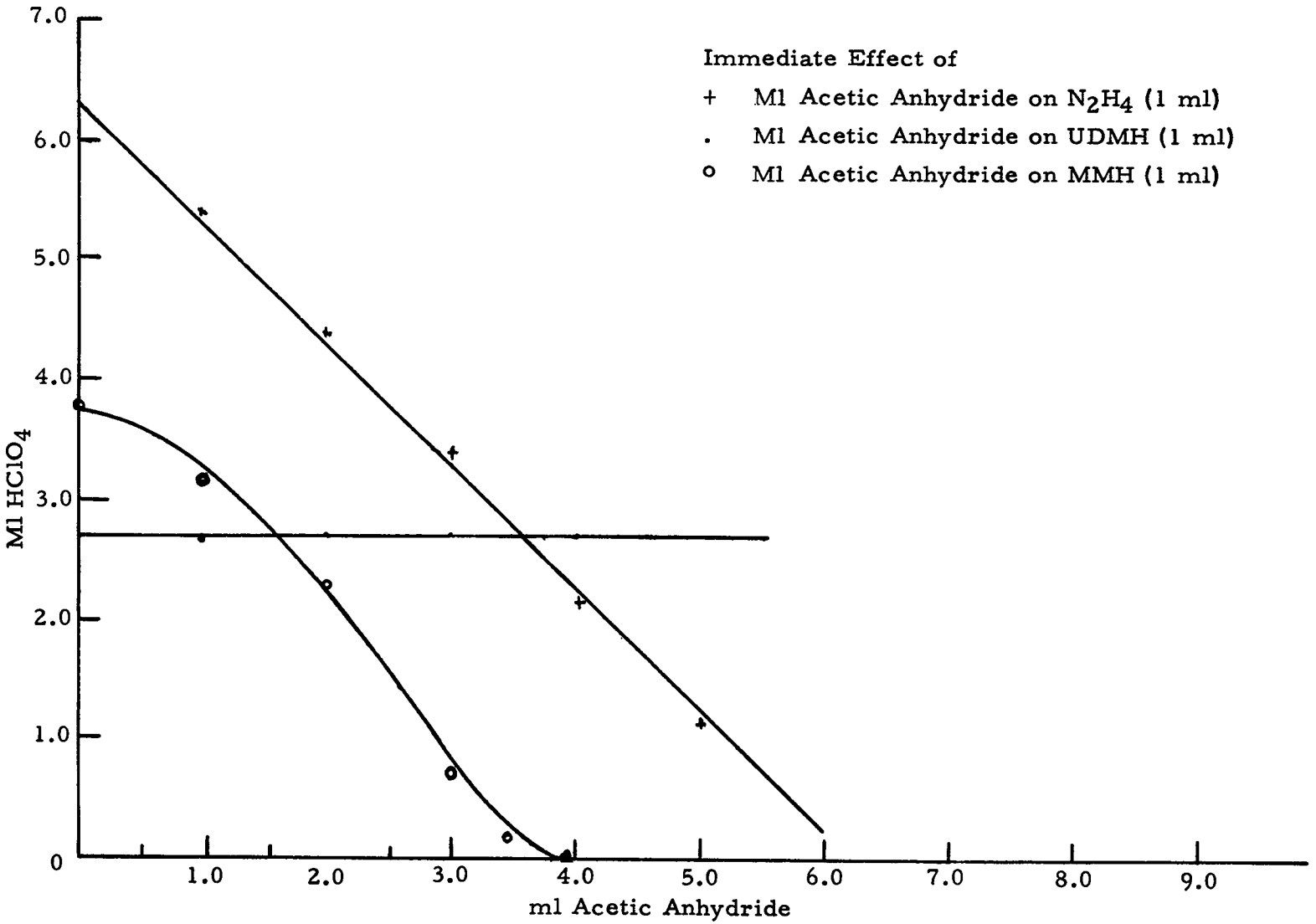


Figure II. The Effect of Acetic Anhydride on Hydrazines

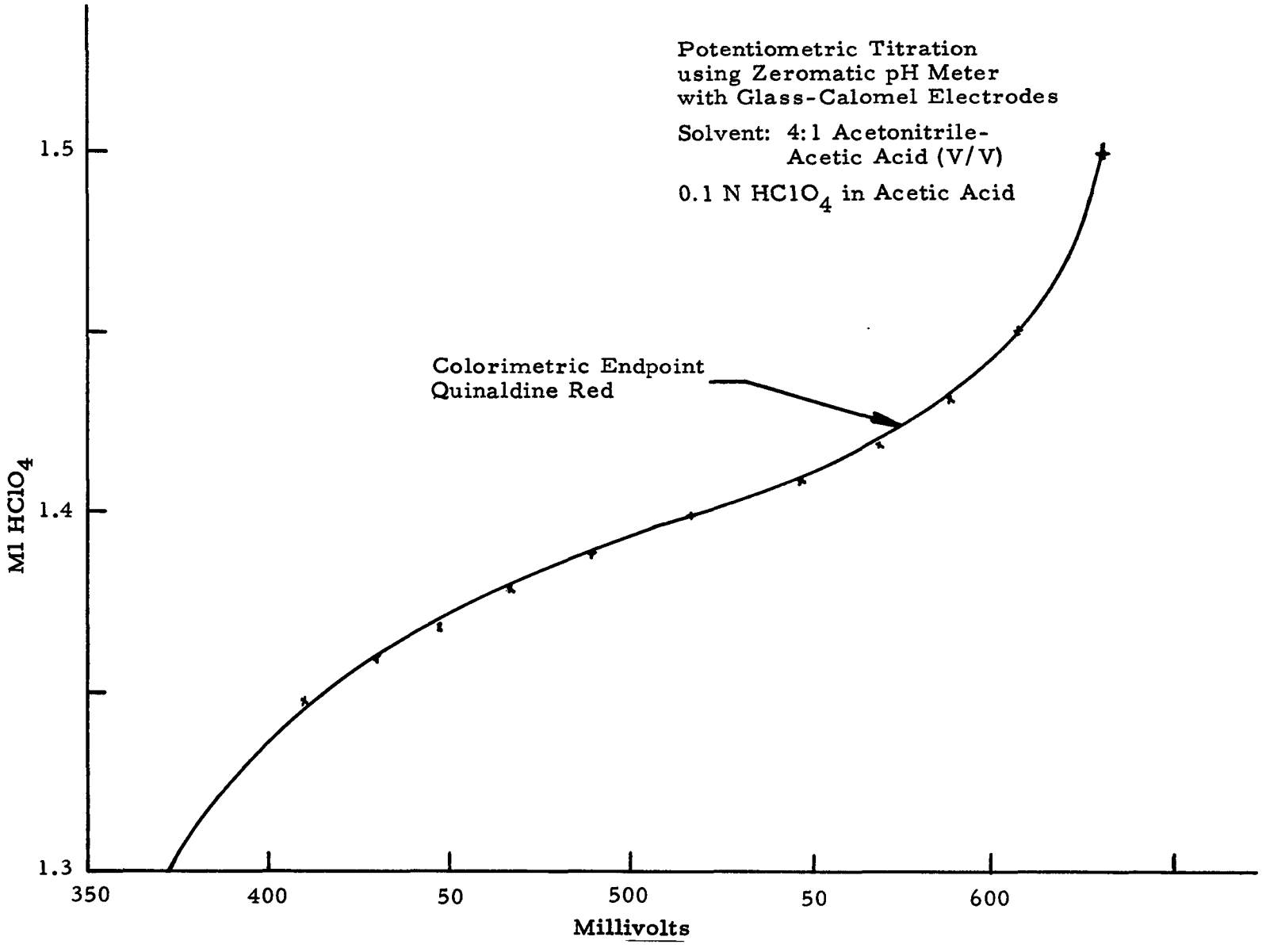


Figure III. Typical Titration Curve for Both Titrations.

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Unclassified Report

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III. In ASTIA collection

acetonitrile-acetic acid medium. Two titrations are required; one measures the total basicity of the hydrazine mixtures; the other titration measures UDMH after the N_2H_4 or MMH has reacted with Ac_2O.

Rocket Research Laboratories, Edwards, Calif. Rpt No. SSD-TDR-62-203. AN ACETYLATION METHOD FOR DETERMINING MIXTURES OF EITHER HYDRAZINE-1, 1-DIMETHYLHYDRAZINE OR MONOMETHYL-HYDRAZINE-1, 1-DIMETHYLHYDRAZINE. 14 p. incl illus. Dec 62.

Unclassified Report

A method is presented for determining mixtures of either hydrazine-1, 1-dimethylhydrazine (N_2H_4/UDMH) or monomethylhydrazine-1, 1-dimethylhydrazine (MMH/UDMH). This method is based on the difference in reaction rates for acetylating hydrazines. Both N_2H_4 and MMH react immediately while the acetylation of UDMH proceeds slowly. The reactions are conducted in

1. Acetylation

I. AFSC Project No. 3148, Task No. 30197

II. Malone, Hugh E., Biggers, R. A.

III. In ASTIA collection

acetonitrile-acetic acid medium. Two titrations are required; one measures the total basicity of the hydrazine mixtures; the other titration measures UDMH after the N_2H_4 or MMH has reacted with Ac_2O.